National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2696

Silica Fume

This Standard Reference Material (SRM) is intended primarily for use in evaluating chemical and instrumental methods of analysis of silica fume used in conjunction with product specifications [1,2]. A unit of SRM 2696 consists of a single bottle containing approximately 70 grams of powder.

The certified values for seven constituents in SRM 2696 are listed in Table 1. Reference values for five constituents and specific surface area are listed in Table 2. For the chemical constituents, values are reported as mass fractions on a dry weight basis [3]. Value assignment categories are based on the definition of terms and modes used at NIST for chemical reference materials [4], and uncertainties are assessed according to the ISO Guide [5]. Analytical methods employed in the development of this material are listed in Table 3.

Certified Values: A NIST-certified value is a value for which NIST has the highest confidence in its accuracy, in that all known or suspected sources of bias have been investigated or accounted for by NIST. A certified value is the present best estimate of the true value based on the results of analyses performed at NIST and cooperating laboratories using the test methods listed in Table 3. The uncertainty listed with the value is an expanded uncertainty (95 % confidence interval [6]) and is calculated according to the method in the ISO/NIST Guides [5].

Reference Values: Reference values are non-certified values that are the present best estimates of the true values. However, the values do not meet the NIST criteria for certification and are provided with associated uncertainties (95 % confidence interval [6]) calculated according to the method in the ISO/NIST Guides [5].

Expiration of Certification: The certification of this SRM is valid until **01 January 2014**, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate, see "Instructions for Use". However, the certification will be nullified if the SRM is damaged or contaminated.

Coordination of the technical measurements for certification was accomplished under the direction of J.R. Sieber of the NIST Analytical Chemistry Division.

Analytical measurements (including homogeneity testing) for certification of this SRM were performed at NIST by J.R. Sieber and A.F. Marlow of the NIST Analytical Chemistry Division.

Statistical consultation for this SRM was provided by S.D. Leigh of the NIST Statistical Engineering Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 06 May 2004

Robert L. Watters, Jr., Acting Chief
Measurement Services Division

Material Preparation: The material for SRM 2696 was obtained from Elkem Materials, Inc.[1] from a typical production batch of silica fume. The material was blended and bottled at NIST under the supervision of M.P. Cronise of the NIST Measurement Services Division.

Caution to Users: In some laboratories, it may be accepted practice to return material to its original container after testing it for Specific Surface Area. Since this SRM is intended for testing of other properties and constituents, the material should be discarded after use. Returning it to the original container may result in contamination of the material and nullification of the certification.

Stability: This material is considered to be stable during the period of certification when stored in its original bottle in a sealed foil pouch. Once the foil pouch has been opened, the moisture content and loss on ignition may be subject to change. NIST will monitor this material and will report any significant changes in certification to the purchaser. Registration (see attached sheet) will facilitate notification.

INSTRUCTIONS FOR USE

Chemical Constituents: To relate analytical determinations to the certified values on this Certificate of Analysis, a minimum sample quantity of 500 mg is recommended. The material must be dried at 110 ºC for at least 2 h, prior to analysis for chemical constituents. The material should be stored in its original container in a cool, dry location.

Moisture Content and Loss on Ignition: The reference values were determined by the cooperating laboratories using the methods of test in ASTM C 311-02 [7]. The use of alternative methods may result in different values for these properties. The determinations were made using material from freshly opened units of the SRM. Once the foil pouch has been opened, the moisture content and loss on ignition may be subject to change.

Specific Surface Area: The reference value provided in Table 2 was determined by the cooperating laboratories using the Brunauer-Emmet-Teller (BET) method of test in ASTM C 1069-86 (1997) [8]. The determinations were performed using both Single-point and Multi-point analyses. The suitability of this SRM for use with different methods and measurement dynamics has not been determined.

Constituent Assignment: The chemical constituents listed in this Certificate of Analysis are expressed as the chemical forms and in the order given in ASTM C 114-99 [9], Section 3, Table 1.

Table 1. Certified Values with Expanded Uncertainties for SRM 2696 Silica Fume

Constituent	Mass Fraction (%)		
SiO_2	95.61	±	0.37[a]
Al_2O_3	0.2080	±	0.0071[a]
CaO	0.426	±	0.016[a]
MgO	0.235	±	0.024[b]
K_2O	0.652	±	0.028[a]
Mn_2O_3	0.032	±	0.004[b]
ZnO	0.051	±	0.005[b]

[a] The certified value is a weighted mean of the results from two to seven analytical methods [10]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-source variance incorporating inter-method bias with a pooled within-source variance following the ISO/NIST Guides [5].

[b] The certified value is an unweighted mean of the results from two to five analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [11] with a pooled, within-method variance following the ISO/NIST Guides [5].

[1] Certain commercial equipment, instruments, or materials are identified in this certificate in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Table 2. Reference Values with Expanded Uncertainties for SRM 2696 Silica Fume

Constituent	Mass Fraction[a] (%)		
Na_2O	0.129	±	0.019[b]
P_2O_5	0.0863	±	0.0057[c]
Fe_2O_3	0.055	±	0.011[b]
Moisture Content	0.251	±	0.034[b]
Loss on Ignition at 750 ºC	2.11	±	0.10[b]
Specific Surface Area (m^2/g)	22.92	±	0.36[b]

[a] Except Specific Surface Area

[b] The reference value is a weighted mean of the results from two to seven analytical methods [10]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-source variance incorporating inter-method bias with a pooled within-source variance following the ISO/NIST Guides [5].

[c] The reference value is an unweighted mean of the results from two to five analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [11] with a pooled, within-method variance following the ISO/NIST Guides [5].

Table 3. Analytical Methods

Constituent	Methods
Na_2O	C 114-99 [9], XRF, ICP-OES, FAAS
MgO	C 114-99, XRF, ICP-OES, FAAS
Al_2O_3	C 114-99, XRF, ICP-OES
SiO_2	C 114-99, XRF
P_2O_5	C 114-99, XRF, ICP-OES
K_2O	C 114-99, XRF, ICP-OES, FAAS
CaO	C 114-99, XRF, FAAS
Mn_2O_3	C 114-99, XRF, ICP-OES
Fe_2O_3	XRF, ICP-OES
ZnO	C 114-99, XRF, ICP-OES
Moisture Content	C 311-02
Loss on Ignition at 750 ºC	C 311-02
Specific Surface Area (m^2/g)	C 1069-86(1997), Single-point and Multi-point analyses, N_2 gas

Key: C 114-99 [9] refers to the classical (wet) chemical methods in this standard
XRF = X-ray fluorescence spectrometry with various sample preparation and calibration procedures
ICP-OES = Inductively-Coupled Plasma Optical Emission Spectrometry
FAAS = Flame Atomic Absorption Spectrophotometry

The Cooperating Laboratories also tested SRM 2696 for oversize in accordance with ASTM C 430. The data generated had a wide variance that did not meet NIST criteria for value assignment. In the minutes of the December 5, 2003 meeting of ASTM International Subcommittee C09.24 Task Group 4 on Silica Fume Specification C1240, the data variance was attributed to the self-agglomerating characteristics of this material and that the measurement of oversize in silica fume requires a modification of ASTM C 430-96(2003) [12].

Cooperating Laboratories: SRM 2696 was produced in cooperation with the Silica Fume Association as Task 11 under Cooperative Agreement DTFH61-99-X00063 between the Federal Highway Administration and the Silica Fume Association. Participation by collaborating laboratories was coordinated by G.M. Gapinski of Norchem, Inc., Ft. Pierce, Florida. Analytical determinations for certification of this SRM were performed by the following laboratories:

G. Gapinski and R. Plotria; Norchem, Inc.; Ft. Pierce, Florida, USA
K. Groff; New York, Dept. Of Transportation, Materials Bureau; Albany, New York, USA
J. Beilman; Kansas Dept. of Transportation; Topeka, Kansas, USA
S. Carlock; Utah Dept. of Transportation, Materials QA Section; Salt Lake City, Utah, USA
D. Broton; Construction Technology Laboratories; Skokie, Illinois, USA
R. Martin; Globe Metallurgical, Inc.; Beverly, Ohio, USA
K. Kasprzak; Globe Metallurgical, Inc.; Niagara Falls, New York, USA
R. Chevrier; CANMET Materials Technology Laboratory; Ottawa, Ontario, Canada
R. Hageman; Elkem Materials Inc.; Alloy, West Virginia, USA
S. Schlorholtz; Iowa State University, Material Analysis Research Laboratory; Ames, Iowa, USA
R. Karuhn; Particle Technology Labs, Inc.; Downers Grove, Illinois, USA
M. Thomas; Quantachrome Corp.; Boynton Beach, Florida, USA
M. Pohl; Horiba Instruments, Inc.; Irvine, California, USA
R. Xu; Beckman Coulter, Inc., Particle Characterization Operation; Miami, Florida, USA

REFERENCES

[1] ASTM C 1240-03a; *Standard Specification for Silica Fume Used in Cementitious Mixtures*; Annu. Book ASTM Stand. Vol. 04.02 (2003).

[2] AASHTO M307-03; *Standard Specification for Use of Silica Fume as a Mineral Admixture in Hydraulic-Cement Concrete, Mortar, and Grout*; Standard Specifications for Transportation Materials and Methods of Sampling and Testing; 23rd ed., American Assoc. of State Highway and Transportation Officials: Washington, DC (2003).

[3] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811, U.S. Government Printing Office: Washington, DC (1995).

[4] May, W.E.; Parris, R.M.; Beck II, C.M.; Fassett, J.D.; Greenberg, R.R.; Guenther, F.R.; Kramer, G.W.; Wise, S.A.; Gills, T.E.; Colbert, J.C.; Gettings, R.J.; MacDonald, B.S.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Spec. Pub. 260-136, U.S. Government Printing Office: Washington, DC; p. 16 (2000).

[5] *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed. ISO, Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at http://physics.nist.gov/Pubs/.

[6] Hahn, G.J.; Meeker, W.Q.; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc., New York (1991).

[7] ASTM C 311-02, *Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use as a Mineral Admixture in Portland-Cement Concrete*; *Annu. Book ASTM Stand.* Vol. 04.02 (2002).

[8] ASTM C 1069-86(1997); *Standard Test Method for Specific Surface Area of Alumina or Quartz by Nitrogen Adsorption*; Annu. Book ASTM Stand. Vol. 03.05 (1997).

[9] ASTM C 114-99, *Standard Test Methods for Chemical Analysis of Hydraulic Cement*; Annu. Book ASTM Stand. Vol. 04.01 (1999).

[10] Ruhkin, A.L.; Vangel, M.G.; *Estimation of a Common Mean and Weighted Means Statistics*; J. Am. Statist. Assoc.; Vol. 93, pp. 303-308 (1998).

[11] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.K.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; *An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol.; Vol. 105, pp. 571-579 (2000).

[12] Minutes, ASTM International Subcommittee C09.24 Task Group 4 on Silica Fume Specification C1240, December 5, 2004.

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751, email srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.